

41F#346

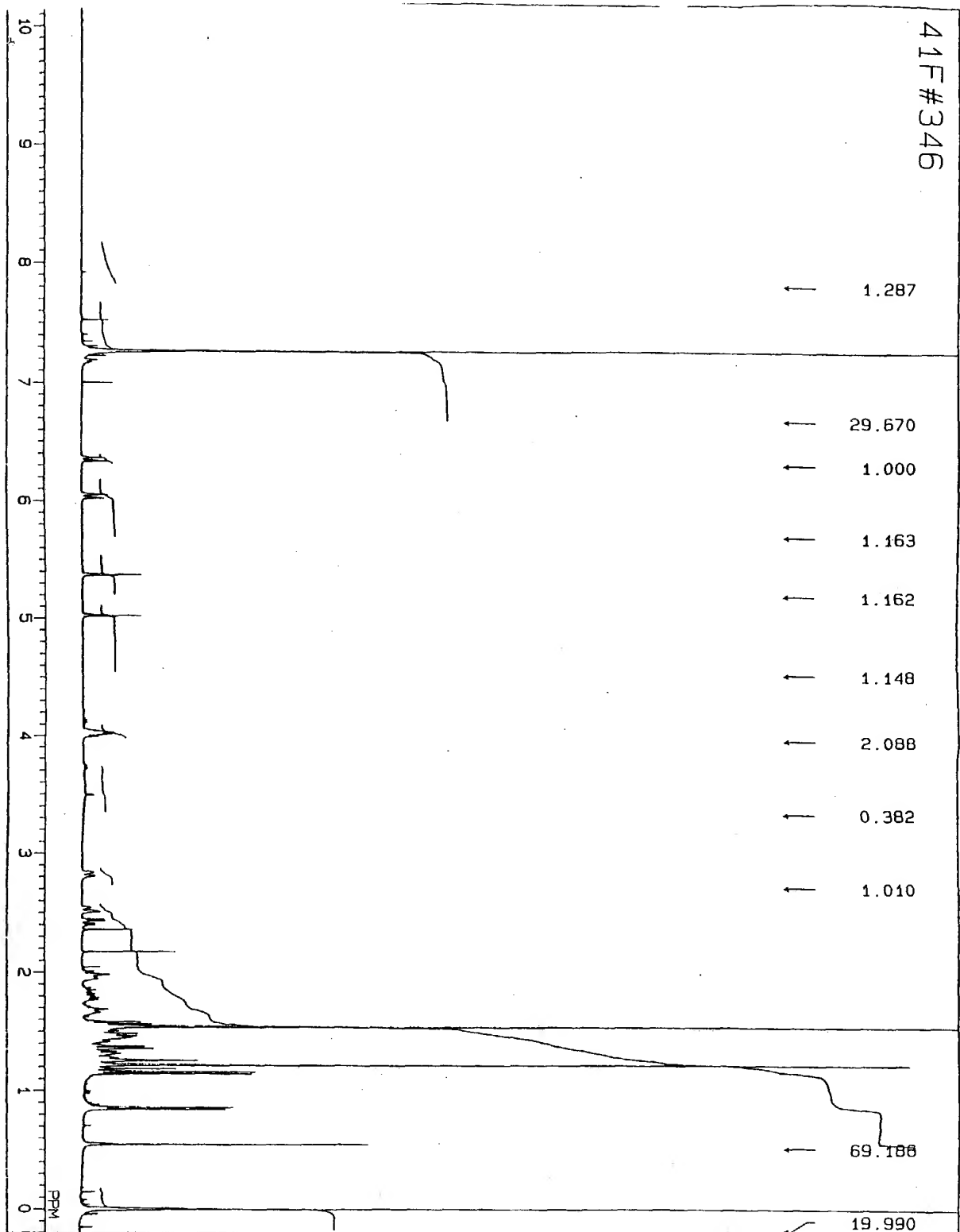


Exhibit 1
Chart 1, p. 1
11:26:05

SLVNT	COCL3
OBNUC	1H
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10905.1 Hz
PM1	5.9 us
POINT	32768
SAMPO	32768
SCANS	9216
DUMMY	0
FREQU	5000.0 Hz
FILTR	5000 Hz
ACQTM	3.277 sec
PD	5.000 sec
RGAIN	25
BF	0.10 Hz
T1	0.0 %
T2	0.0 %
T3	90.0 %
T4	100.0 %
EXMOD	SGNON
DFILE	(100.140)FN0346
SHMFL	TH5
SPEED	15 Hz
OPERATOR	J.SHIWODE

MAIN				Secondary	
RESOL				0.000000 Hz	
Error				0.000000 Hz	
DBS				-2398.36 Hz	
NMAIN				AB085-599764, 9000000 Hz	
				23	
NMAIN				41F4346	
NO.	FTN	INT (°)	FREQ (MHz)	POSITION	BAR GRAPH
1	7.91370	0.07937	316.76	3684	0
2	7.51961	0.48464	3004.79	6200	0
3	7.40222	0.05175	2951.75	6354	0
4	7.39382	0.12111	2925.93	6365	0
5	7.39149	0.20583	2935.18	6365	0
6	7.24007	100.00000	2902.53	6365	0
7	7.23283	0.38801	2878.66	6365	0
8	7.22057	0.43407	2868.66	6365	0
9	7.18449	0.28503	2827.31	6439	0
10	7.17359	0.19619	2849.87	6447	0
11	7.15589	0.17827	2864.79	6523	0
12	7.15589	0.17827	2864.79	6523	0
13	7.13899	0.04443	2854.00	6499	0
14	6.99614	0.53732	2756.94	6866	0
15	6.93951	0.41519	2552.42	7720	0
16	6.31226	0.49927	2351.13	7157	0
17	6.08700	0.48505	2187.66	8129	0
18	5.91705	0.32527	2106.31	8166	0
19	5.91705	0.32527	2106.31	8166	0
20	5.91705	0.32527	2106.31	8166	0
21	5.34944	1.04497	2146.61	9017	0
22	5.30465	0.63816	2124.78	9023	0
23	5.02593	0.59430	2009.28	9467	0
24	5.02135	1.05655	2007.45	9473	0
25	5.01753	0.78797	2005.92	9478	0
26	4.81128	0.08595	1651.61	10439	0
27	4.70518	0.12551	1619.87	10743	0
28	4.04349	0.23001	1616.52	10754	0
29	4.03128	0.35895	1611.63	10770	0
30	4.01601	0.54112	1605.53	10790	0
31	4.00837	0.43187	1603.48	10800	0
32	3.79311	0.28451	1596.37	10820	0
33	3.72470	0.18413	1481.85	11152	0
34	3.72212	0.10795	1486.04	11175	0
35	3.70914	0.09723	1484.85	11172	0
36	3.69156	0.05912	1475.83	11215	0
37	3.48722	0.30733	1399.79	11467	0
38	3.48722	0.30733	1399.79	11467	0
39	3.48722	0.30733	1399.79	11467	0
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80	3.48722	0.30733	1399.79	11467	0
81	3.48722	0.30733	1399.79	11467	0
82	3.48722	0.30733	1399.79	11467	0
83	3.48722	0.30733	1399.79	11467	0
84	3.48722	0.30733	1399.79	11467	0

50	2.3646	0.2507	957.36	12914
51	2.3971	0.9189	941.77	12965
52	2.1231	1.7066	868.53	13005
53	2.00518	0.3157	817.87	13172
54	2.00518	0.0586	813.61	13362
55	2.00518	0.0586	813.61	13502
56	1.59167	0.5010	792.24	13452
57	1.53724	0.2813	782.47	13487
58	1.94350	0.2859	776.96	13505
59	1.88249	0.05586	723.78	13581
60	1.88249	0.18540	746.15	13606
61	1.88249	0.1716	742.45	13618
62	1.88249	0.1681	735.47	13641
63	1.82394	0.1881	725.96	13659
64	1.81725	0.25048	726.42	13670
65	1.80762	0.22568	722.66	13683
66	1.80075	0.24723	719.91	13692
67	1.79063	0.2017	715.74	13700
68	1.78394	0.22445	713.20	13714
69	1.77898	0.23447	710.75	13722
70	1.77898	0.23447	708.79	13732
71	1.76793	0.29629	704.06	13744
72	1.76106	0.22537	697.02	13767
73	1.74500	0.07765	691.80	13803
74	1.74500	0.07765	691.80	13803
75	1.74500	0.17777	664.92	13878
76	1.62904	0.29028	664.92	13878
77	1.65953	0.34395	664.92	13878
78	1.65953	0.34395	664.92	13878
79	1.62842	0.13878	649.73	13923
80	1.59770	0.10947	638.73	13958
81	1.57938	1.06582	631.41	13982
82	1.56411	1.27070	625.31	14002
83	1.51256	0.63027	615.75	14031
84	1.51256	0.63027	615.75	14031
85	1.50305	0.67469	600.89	14082
86	1.48071	1.00431	598.07	14111
87	1.45077	0.78544	589.07	14124
88	1.44579	0.98350	583.19	14140
89	1.44579	0.98350	583.19	14140
90	1.44579	0.98350	583.19	14140
91	1.43892	0.74082	575.26	14164
92	1.43892	0.44455	568.85	14187
93	1.41536	0.50507	565.80	14197
94	1.40839	0.35961	563.05	14206
95	1.41232	0.28848	559.39	14218
96	1.41232	1.29746	542.40	14272
97	1.41232	1.29746	542.40	14272
98	1.33205	0.63017	532.52	14300
99	1.31602	0.70781	526.13	14327
100	1.29594	0.48537	518.49	14352
101	1.28946	0.58335	513.31	14369
102	1.27406	0.52894	510.96	14377
103	1.26259	0.48537	504.76	14377
104	1.26259	0.48537	504.76	14377
105	1.25954	1.14619	503.94	14401
106	1.25343	2.09311	501.10	14409
107	1.24589	0.97872	498.05	14479
108	1.23589	0.43210	493.16	14435
109	1.22247	0.57958	490.72	14443
110	1.21630	1.46905	487.02	14457
111	1.18320	1.46905	487.02	14457
112	1.15725	3.12907	465.62	14535
113	1.14045	3.06572	455.93	14537
114	1.11602	0.19707	449.17	14581
115	1.07633	0.09577	450.30	14619
116	1.05248	0.11334	422.36	14647
117	1.01297	0.07260	404.93	14722
118	1.01297	0.07260	404.93	14722
119	0.97694	0.18018	398.26	14745
120	0.98015	0.16783	391.85	14767
121	0.93699	0.07737	375.67	14820
122	0.92061	0.10616	366.04	14845
123	0.911457	0.09463	364.38	14857
124	0.89618	0.10850	358.29	14877
125	0.88901	2.73067	351.62	14927
126	0.88199	2.58626	343.61	14948
127	0.87499	2.44199	336.61	14969
128	0.70687	0.16637	282.59	15125
129	0.70435	5.15249	218.51	15335
130	0.50763	0.05559	205.94	15386
131	0.50763	0.05559	205.94	15386
132	0.08168	0.03834	38.29	15800
133	0.08168	0.03834	38.29	15800
134	0.00753	1.92187	16.48	15941
135	0.00000	67.77723	0.00	16031
136	-0.00840	2.61810	3.36	16062
137	-0.00042	0.28183	-16.17	16104
138	-0.00042	0.28183	-16.17	16104
139	-0.18462	0.17588	-57.81	16247

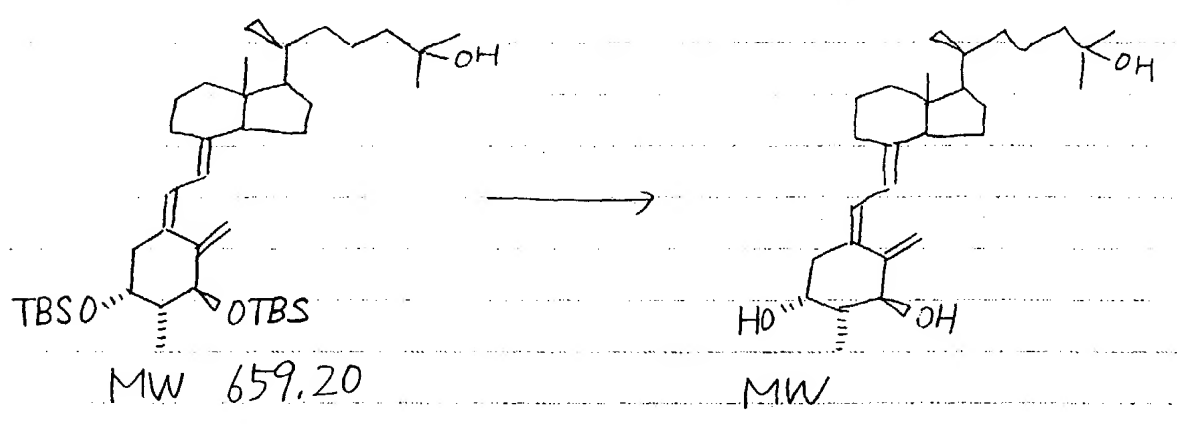
1373

446

Exhibit 1
Chart 1, p.2

4.5

#346



§ #345のwork up

CSA
MeOH

11 mg

1 me

20:30~

1時間後は反応液から MeOH を

とは"1. 水を加え EA抽出

11:00

brine 洗水. $MgSO_4$ 上 脱水

そのエピソード。

シリカゲルカラムで分離後 ~~2HAc~~

HPLC カラム (ODS #18) で分離佳.

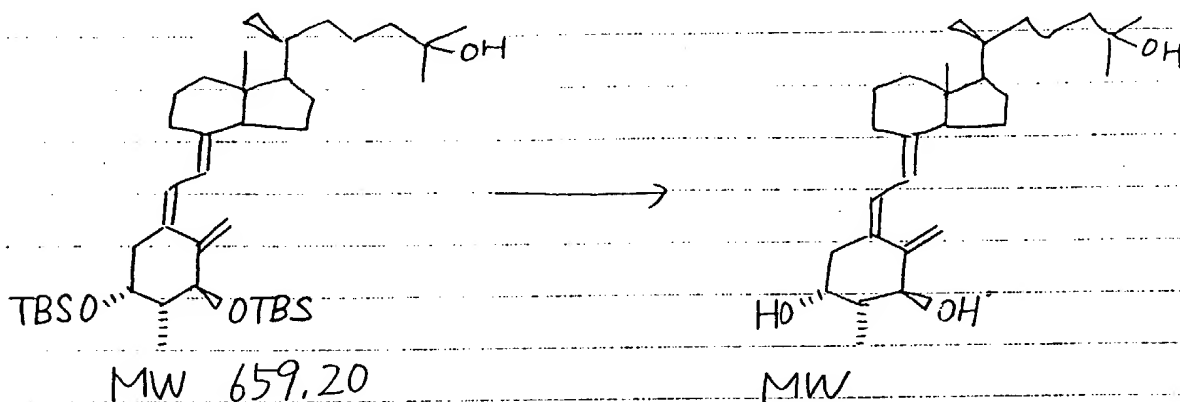
RP-18

4.5mg

(4.31%)

4.5

#346



#345のworkup
CSA
MeOH

11mg
1ml

20:30~

rtかくはん後 反応液から MeOH を
とほし 水を加え EA 抽出
brine 洗い MgSO₄ 上 脱水
ろか、エバポ

11:00

シリカゲルカラムで分離後 ~~エバポ~~ 4.5mg
HPLC カラム (ODS (18)) で分離 (y. 31%)
RP-18

After stirring at rt, MeOH was evaporated from reaction mixture,
water was added and extracted with EA
washed with brine, dried over MgSO₄
filtered, evaporated

After separation by silica gel column chromatography
separation by HPLC column (ODS (18))

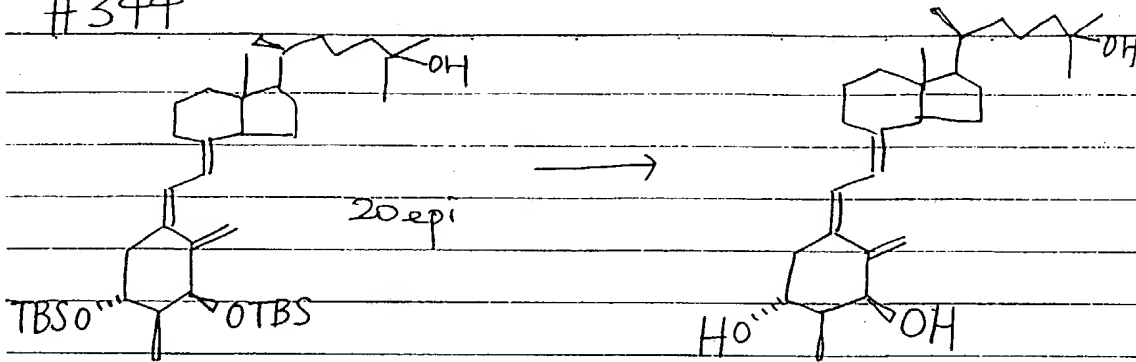
33.8904
9206

33.5847
594

30.2

10 mg

#344



MW

MW 430.67

#343 本體 work up

CSA MW 232.30 11 mg
MeOH 1 ml

AcF 14:20 ~
~50 ml

9:00

MeOHを留去し、水を加え、EA抽出、brine洗い。

MgSO₄上脱水。ろか！エバポレート。

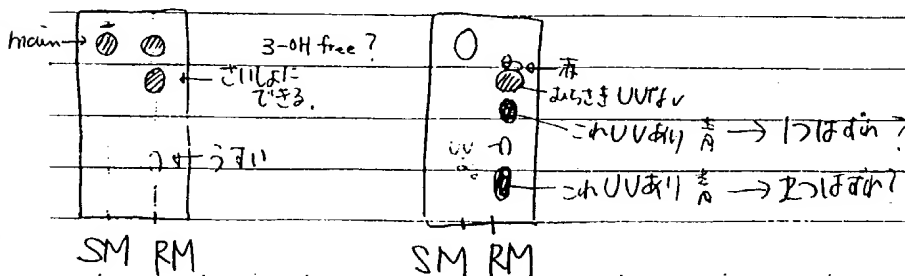
シリカゲルカラム (Φ0.9 cm 10 cm height, EA=n-hex=1:1)

にて精製

9.3 mg (y. 63%)

→ HPLCにて分離。

EA=n-hex=1:1



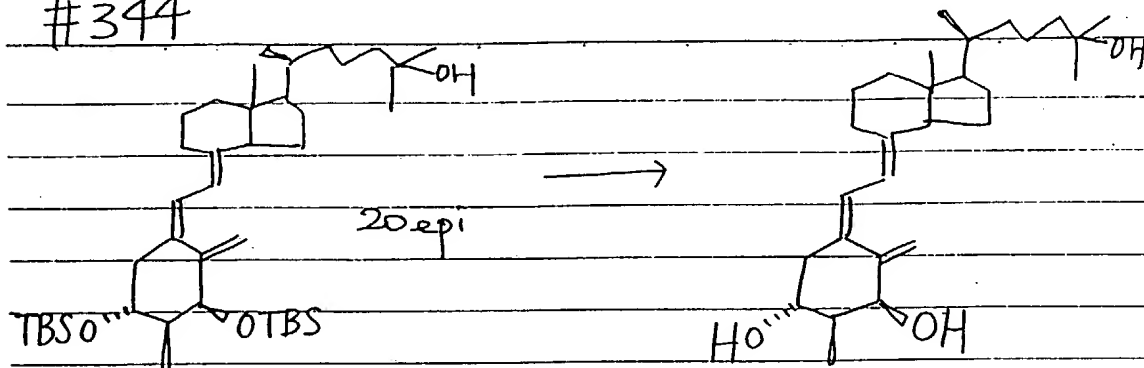
33.8904
9225

33.5847
594

60 mg

No.
cur

#344



MW

MW 430.67

[protective material]

{ #343 [木]体 work up

CSA MW 232.30

11 mg

stirring under Ar at rt

MeOH 1 ml

[Ar F 1/2 1/2] 14:20 ~

9:00

~50 ml

MeOHを留去し、水を加え、EA抽出、brine洗う。

MgSO₄上脱水、ろ過、エバポレート。

シリカゲルカラム (φ0.9 cm 10 cm height, EA=n-hex = 1:1)

にて精製

9.3 mg (y. 63%)

→ HPLCにて分離

MeOH was distilled away, water was added, extracted with EA, washed with

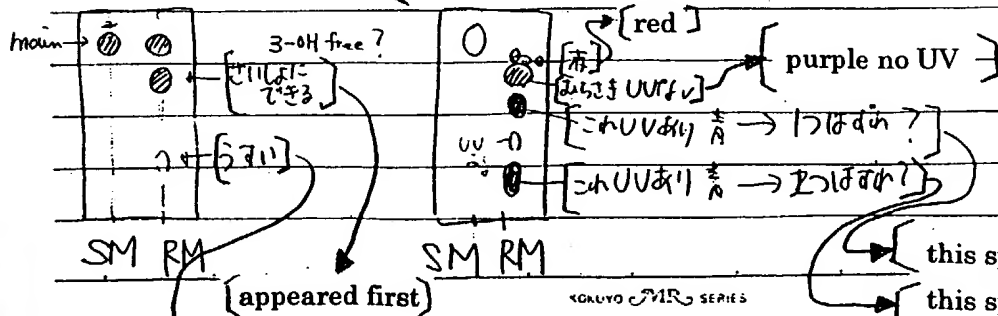
dried over MgSO₄, dehydration, filtration, evaporation

purification by silica-gel column chromatography

(φ0.9 cm 10 cm height, EA/n-Hex = 1:1)

→ Separation by H

EA=n-hex=1:1



KOYO TMR SERIES